Air



NESHAP - Glass Manufacturing Arsenic

Emission Test Report Indiana Glass Company Dunkirk, Indiana



SUMMARY TEST REPORT

STANDARDIZATION AND VALIDATION OF METHODOLOGY TO MEASURE INORGANIC ARSENIC EMISSIONS FROM STATIONARY SOURCES
Indiana Glass Company Dunkirk, Indiana

by

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CONTENTS

		<u>Page</u>
Figu: Table Ackne		iv vi vii
1.	Introduction	1-1
2.	Summary and Discussion of Test Results	2-1
	 2.1 Sampling and analytical protocol 2.2 Test resultselevated temperature runs 2.3 Method 108 traverse test results 2.4 Process samples 	2-1 2-2 2-15 2-18
3.	Project Quality Assurance	3-1
4.	Sampling Location and Test Methods	4-1
	4.1 Sampling and analytical procedures	4-7
5.	Process Operation	5-1
Appe	ndices	
A B C D E	Computer Printouts and Example Calculations Field Data Laboratory Data Sampling and Analytical Procedures Equipment Calibration Procedures and Results Process Description and Operation During Testing	A-1 B-1 C-1 D-1 E-1 F-1

FIGURES

Number		Page
2-1	Quad Train System for Elevated Temperature Tests	2-3
3-1	Pre-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-1, Train A)	3-6
3-2	Pre-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-5, Train B)	3-7
3-3	Pre-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-8, Train D)	3-8
3-4	Pre-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-10, Train C)	3-9
3-5	Pre-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-9, Single Point-Traverse Tests)	3-10
3-6	Pre-Test Thermocouple Digital Indicator Audit Data Sheet (Indicator No. 220)	3-11
3-7	Pre-Test Thermocouple Digital Indicator Audit Data Sheet (Indicator No. 221)	3-12
3-8	Pre-Test Onsite Audit Data Sheet	3-13
3-9	Pre-Test Onsite Audit Data Sheet	3-14
3-10	Mid-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-1)	3~15
3-11	Mid-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-5)	3-16
3-12	Mid-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-8)	3-17
3-13	Mid-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-10)	3-18

FIGURES (continued)

Number		Page
3-14	Mid-Test Thermocouple Digital Indicator Audit Data Sheet (Indicator No. 220)	a 3-19
3-15	Mid-Test Thermocouple Digital Indicator Audit Data Sheet (Indicator No. 221)	a 3-20
3-16	Mid-Test Onsite Audit Data Sheet	3-21
3-17	Mid-Test Onsite Audit Data Sheet	3-22
3-18	Post-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-1)	1 3-23
3-19	Post-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-5)	1 3-24
3-20	Post-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-8)	1 3-25
3-21	Post-Test Audit Report: Dry Gas Meter by Critical Orifice (Meter Box FB-11)	1 3-26
3-22	Post-Test Thermocouple Digital Indicator Audit Da- Sheet (Indicator No. 220)	ta 3 - 27
3-23	Post-Test Thermocouple Digital Indicator Audit Da- Sheet (Indicator No. 221)	ta 3-28
3-24	Example of Unacceptable Dry Gas Meter Audit	3-29
3-25	Example of Onsite Calibration Data Sheet	3-31
4-1	Quad Train System for Elevated Temperature Tests	4-2
4-2	Four-Train Sampling System Showing Nozzle, Pitot Tube, and Thermocouple Position	4-3
4-3	Furnace Exit Stack Elevation	4-5
4-4	Furnace Exit Stack Sampling Port Location	4-6

TABLES

Number		Page
2-1	Summary of Sample Conditions	2-4
2-2	Summary of Arsenic Analytical Results - Quad and Reference Train Runs	2-5
2-3	Statistical Data for Grouped Runs	2-7
2-4	Statistical Data for Grouped Runs - EMSL Quad Train Tests	2-10
2-5	Summary of Sample and Flue Gas Conditions Arsenic Traverse Tests	2-16
2-6	Summary of Arsenic Analytical Results - Traverse Train	2-17
2-7	Process Sample Analytical Results	2-19
3-1	Field Equipment Calibration	3-3
3-2	Arsenic Blank Data	3-32
3-3	Arsenic Laboratory Reagent Blank Data	3-34
3-4	Linear Regression Data (Flame)	3-36
3-5	Arsenic Audit Results	3-37
3-6	Arsenic Standard Addition Results	3-39
3-7	Linear Regression Data (Furnace)	3-41
3-8	Duplicate Analysis Data	3-43

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Mr. Daniel Bivins, EPA-EMB Task Manager, provided overall project coordination and guidance and observed the test program.

Mr. Ronald Myers, EPA lead engineer, Industrial Studies Branch, provided project coordination relative to process operation and overall project scope. Mr. Larry Keller, representing Radian Corporation (an EPA contractor) monitored process operation throughout the test period. Mr. Charles Bruffey was the PEI Project Manager. Principal authors were Messrs. Charles Bruffey and Thomas Wagner.

SECTION 1

INTRODUCTION

Arsenic is listed as a hazardous air pollutant under Section 112 of the Clean Air Act (National Emission Standards for Hazardous Air Pollutants). To protect public health from unreasonable risks associated with exposure to airborne arsenic, the U.S. Environmental Protection Agency (EPA) has developed standards to decrease inorganic arsenic emissions from the following source categories: high-arsenic primary copper smelters, low-arsenic primary copper smelters, and glass manufacturing plants.

To support the standards review process and provide additional arsenic emissions data from glass manufacturing facilities, PEI Associates, Inc., under contract to Research Triangle Institute and directed by the Source Branch of the EMSL Quality Assurance Division and the Emission Standards and Engineering Division - Emission Measurement Branch, performed a series of atmospheric emission tests on a glass melting furnace at Indiana Glass Company in Dunkirk, Indiana. These tests were conducted from May 17 through 19, 1984, as part of a larger study designed to evaluate the sampling and analytical procedures for measuring inorganic arsenic from stationary sources. Proposed Method 108* provides total arsenic results (particulate plus gaseous fraction).

⁴⁰ CFR 61, Appendix B, Method 108, July 1983.

The primary objective of this test program was to determine the precision of proposed Method 108. Relative standard deviations (the standard deviation expressed as a percent of the mean value) of four-train (quad) sample runs were used to estimate method precision. A total of nine quad train runs representing 36 individual samples were conducted using Method 108 sampling and analytical procedures as described in the Quality Assurance Project Plan developed and submitted in January 1984 to the EPA Environmental Monitoring Systems Laboratory. These data are summarized in a report issued to EMSL-QAD.

In this specific portion of the test program, four quadtrain tests were conducted using Method 108 procedures except that probe and filter temperatures were elevated to approximately 204°C and 288°C in order to evaluate the effects of increased sampling train temperature on arsenic distribution in the sampling train. During these runs, a single Method 108 sampling train (121°C) was run for reference purposes. Three Method 108 traverse tests were also conducted to provide additional data in support of the arsenic standards developed to date.

Section 2 summarizes and discusses the test results; Section 3 addresses quality assurance considerations specific to this project; Section 4 describes the sampling locations and test procedures; and Section 5 describes source operation. Appendix A presents sample calculations and computer printouts; Appendices B and C contain the field data sheets and laboratory analytical

results, respectively; Appendix D details the sampling and analytical procedures; Appendix E summarizes equipment calibration procedures and results; and Appendix F contains a process description and the furnace operating data for the test period.

SECTION 2

SUMMARY AND DISCUSSION OF TEST RESULTS

2.1 SAMPLING AND ANALYTICAL PROTOCOL

A four-train (quad) sampling system was used to collect samples at the furnace exit stack. This system allows four trains to sample simultaneously at essentially a single point in the stack (see Section 4).

Because this sampling approach allows simultaneous sampling at essentially a single point, it reduces the effect of variations in the velocity and particulate profiles on the sampling results. It also permits a statistically significant number of samples to be taken in a short amount of time. Further, since two of the four trains are identical for every run, the withintrain precision can be determined at the same time as the relationship of the different trains is being compared.

The Quad runs conducted were designed to evaluate the effect of arsenic collection at elevated sampling temperatures. Two of the trains were heated to approximately 204°C (400°F) and two trains were heated to approximately 288°C (550°F) for comparative purposes. Additionally, in three of the four quad tests conducted, backup filters were maintained at approximately 121°C (250°F) prior to the impinger section.

Figure 2-1 depicts the quad train configuration used for these tests. Individual train components were recovered and analyzed for arsenic separately to evaluate the distribution of arsenic in the sampling train. In each train, the contents of the first and second impingers were recovered, combined, and analyzed for arsenic and the third and fourth impingers were recovered, combined, and analyzed for arsenic. The probe rinse and front filter were recovered and analyzed according to procedures defined in Method 108. In Trains A and D, the back-half glassware of the front filter, the glass connector, and the front-half glassware of the backup filter were rinsed with 0.1 N NaOH and this rinse was analyzed for arsenic. The backup filter was analyzed separately in each case.

During these runs, a single Method 108 sampling system (designated RT) (121°C) was run for reference purposes. Three multipoint traverse tests utilizing a single Method 108 train were also conducted at the completion of the quad-train tests.

In each train, the probe and filter temperatures were set at a predetermined temperature and monitored using multiterminal digital indicators with thermocouple leads located in each probe and immediately behind the Method 5 filter frits.

2.2 TEST RESULTS--ELEVATED TEMPERATURE RUNS

Table 2-1 summarizes sampling conditions for the quad train, reference train, and traverse train (designated CD) test runs.

Table 2-2 summarizes the arsenic analytical results by sample

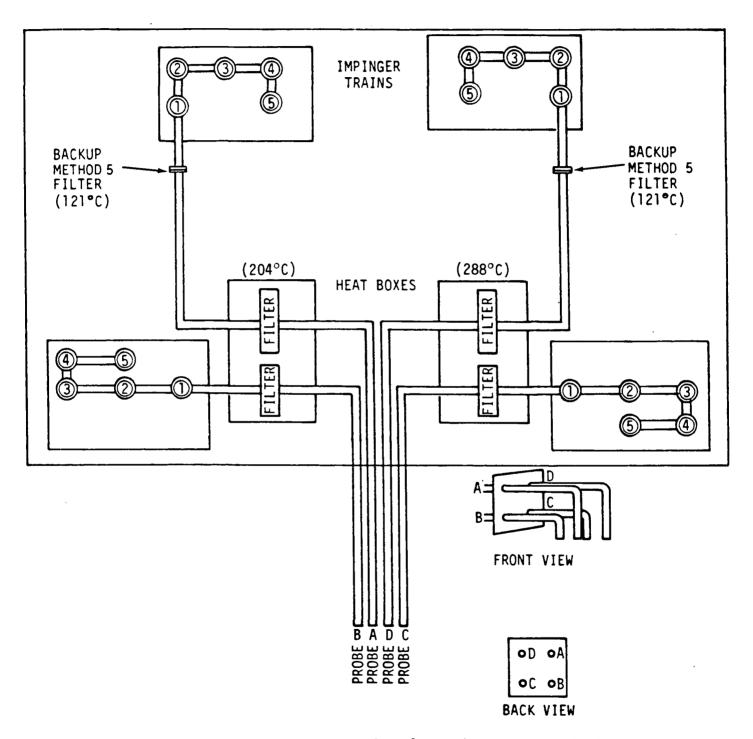


Figure 2-1. Quad train system for elevated temperature tests.

TABLE 2-1. SUMMARY OF SAMPLE CONDITIONS

						Sampling con	ditions		
Run No.	Sampling type	Date (1984) and time (24-h)	Metered volume, dsm ³	Isoki- netic,%	Mois- ture,%	Gas temper- ature, °C	Probe temper- ature, °C	Filter temper- ature, °C	Backup fil- ter temper- ature, °C
10A 10B 10C 10D	Modified Method 108	5/17 11:45-12:55	1.07 1.12 1.18 1.09	101.0 97.5 101.2 100.4	8.4 8.9 8.4 8.6	298 298 298 298	208 206 248 178	204 206 284 286	91 NA NA 120
10-RT	Method 108	5/17 11:45-12:55	1.41	96.0	8.1	301	121	121	NA
11A 11B 11C 11D	Modified Method 108	5/17 17:11-18:21	1.02 1.12 1.14 1.00	101.2 97.8 100.2 99.3	8.5 6.9 8.3 8.8	301 301 301 301	193 200 295 286	201 210 262 285	NA NA NA NA
11-RT	Method 108	5/17 17:11-18:21	1.33	98.7	8.4	311	121	124	NA
12A 12B 12C 120	Modified Method 108	5/18 10:59-12:09	1.23 1.33 1.34 0.29	107.1 103.0 101.2 101.2	8.6 8.4 8.6 9.1	286 286 286 286	205 210 279 174	208 210 261 284	118 NA NA 104
12-RT	Method 108	5/18 10:59-12:09	1.53	99.8	8.5	292	121	122	NA
13A 13B 13C 13D	Modified Method 108	5/18 15:36-16:46	1.14 1.28 1.30 1.13	94.5 98.2 101.4 100.0	8.9 8.7 8.6 9.7	274 274 274 274	208 209 281 279	206 209 239 263	122 NA NA 121
13-RT	Method 108	5/18 15:36-16:46	1.37	100.6	9.0	301	121	122	NA
CD-1	Method 108	5/19 10:32-11:45	1.03	105.6	8.0	260	147	143	NA
CD-2	Method 108	5/19 13:05-14:18	1.06	102.1	8.3	255	188	124	NA
CD-3	Method 108	5/19 15:12-16:22	1.07	100.3	7.7	248	180	123	NA

TABLE 2-2. SUMMARY OF ARSENIC ANALYTICAL RESULTS
QUAD AND REFERENCE TRAIN RUNS

====					Arsenic san	nple weigh	its, mg						
						Back-haìf ^b					Concentration, mg/dsm³		
Run No.	Sample volume, dNm³	Probe rinse	Front ^a filter	Total front half	Glass connector	Backup filter	Impingers 1 & 2	Impingers 3 & 4	Total back half	Front half	Back half	Total train	
10A	1.07	0.323	8.50	8.82	0.05	0.05	0.05	0.05	0.05	8.24	-	8.24	
10B	1.12	0.686	9.26	9.95	NA	NA	0.05	0.05	0.05	8.88	-	8.88	
10C	1.18	0.483	9.81	10.29	NA NA	NA	0.05	0.05	0.05	8.72	-	8.72	
10D	1.09	0.775	9.81	10.59	0.05	0.05	0.05	0.05	0.05	9.72	-	9.72	
10-RT	1.41	1.09	11.17	12.26	NA NA	NA	0.05	NA	0.05	8.70	-	8.70	
11A	1.02	0.792	7.57	8.36	NA NA	NA	0.05	0.05	0.05	8.20	-	8.20	
11B	1.12	0.666	9.19	9.86	NA NA	NA	0.05	0.05	0.05	8.80	-	8.80	
110	1.14	0.618	8.98	9.60	NA NA	NA	0.05	0.05	0.05	8.42	-	8.42	
11D ^C	1.00	0.468	1.75	2.22	ΝΩ	NA NA	2.38	0.21	2.59	2.72	2.59	4.81	
11-RT	1.33	1.21	11.17	12.38	NA NA	NA.	0.05	0.05	0.05	9.31	-	9.31	
12A	1.23	0.280	10.48	10.76	0.05	0.05	0.05	0.05	0.05	8.75	-	8.75	
12B	1.33	0.786	10.86	11.65	NA NA	NA NA	0.05	0.05	0.05	8.76	-	8.76	
12C	1.34	0.490	10.96	11.45	NA NA	NA NA	0.05	0.05	0.05	8.54	-	8.54	
120 ^d	0.292	0.627	2.05	2.68	0.05	0.05	0.05	0.05	0.05	9.18	-	9.18	
12-RT	1.53	1.11	10.79	11.90	NA NA	NA NA	0.05	0.05	0.05	7.78	-	7.78	
13A	1.14	0.926	9.26	10.19	0.05	0.05	0.05	0.05	0.05	8.94	-	8.94	
138	1.28	0.866	10.55	11.42	NA NA	NA	0.05	0.05	0.05	8.92	-	8.92	
13C	1.30	0.845	10.35	11.20	NA NA	NA NA	0.05	0.05	0.05	8.62	-	8.62	
13D	1.13	0.786	9.55	10.34	0.05	0.05	0.05	0.05	0.05	9.15	-	9.15	
13-RT	1.37	1.31	11.50	12.81	NA NA	NA	0.10	NA	0.10	9.35	0.07	9.42	

^aThe front filter data include the Parr bomb results, which constituted approxmately 1 percent of the total arsenic on the filter.

 $^{^{}b}$ NaOH rinse and impinger solution blank values ranged from 0.0 to 0.05 mg, therefore, back half values less than 0.05 mg are not reported.

c_{Run} 11D is considered void due to a ruptured filter frit and subsequent loss of sample (see Pages 2-8 and 2-12).

d_{Run} 12D was terminated approximately 14 minutes into the test due to a ruptured filter frit support (see Page 2-12).

fraction, and Table 2-3 presents statistical data for the grouped quad runs.

Sample volumes were consistent and ranged between 1.00 and 1.34 dsm3 for the guad runs conducted during the full test period. Quad Run 12D was terminated approximately 14 minutes into the test due to a broken filter frit; the sample volume for this run was 0.292 dsm3. Sample volumes for the reference train tests ranged between 1.33 and 1.53 dsm3. Isokinetic sampling rates ranged from 96.0 to 107.1 percent, which is within the acceptable range of 90 to 100 percent. The probe and filter temperatures represent average values determined from data recorded on the field data sheets. The desired temperature for paired Trains A and B was 204°C and for paired Trains C and D, 288°C. As shown, filter temperatures for Trains A and B ranged from 201°C to 210°C and the probe temperatures ranged between 193° and 210°C. In Trains C and D, the filter temperatures were more variable ranging between 239° and 286°C, and the probe temperatures ranging from 174° to 295°C. The backup filter temperatures in Runs 10A and 10D, 12A and 12D, and 13A and 13D ranged from 91° to 122°C. No backup filters were utilized for Quad Run 11. In each quad test, the reference train probe and filter temperature was maintained at approximately 121°C.

The moisture content of the stack gas was generally consistent in each run, and the average gas temperatures ranged from 274° to 310° C.

As shown in Table 2-2, arsenic sample weights are reported in milligrams (mg) for each sample fraction analyzed. The front

TABLE 2-3. STATISTICAL DATA FOR GROUPED RUNS

Quad Run No.	Individual run value	Group _b mean	σ, mg/dsm³	RSD, ^d %
10A 10B 10C 10D	8.24 8.88 8.72 9.72	8.89	0.617	6.9
11A 11B 11C 11D	8.2 8.80 8.42 4.81	8.47	0.304	3.6
12A 12B 12C 12D	8.75 8.76 8.54 9.18	8.68	0.124	1.4
13A 13B 13C 13D	8.94 8.92 8.62 9.15	8.91	0.218	2.4
Overall means		8.74 ^e	0.367 ^f	4.20 ^g

 $^{^{\}mathrm{a}}$ Sample Nos. 11D and 12D are considered invalid and are not included in the group data.

$$9_{\overline{RSD}} = \sqrt{\frac{\Sigma \sigma^2}{n}/\overline{\lambda}}$$
.

^bMean concentration.

^CWithin-run standard deviation with N-1 weighting for sample data.

 $^{^{\}mbox{\scriptsize d}}\mbox{\sc Within-run}$ relative standard deviation is the standard deviation expressed as a percent of the mean concentration.

 $^{^{\}mathbf{e}}$ Simple averages of tabulated data.

fPooled standard deviation; $\sqrt{\frac{\Sigma \sigma^2}{n}}$.

filter weight includes results for both the NaOH extract and the Parr bomb (HF/HNO₃) extract. The extract results constituted approximately 1 percent of the total arsenic on the filter.

Arsenic was found mainly in the front half (probe and filter) of each of the 16 individual trains with the exception of Run 11D. During this run, the filter frit support ruptured but sampling continued until completion of the run. Obviously, some arsenic was carried to the back half of the sampling train as evidenced by the reported weight (2.59 mg) in the back half. Run 11D is considered an invalid sample and is not included in any of the grouped averages or statistical calculations.

The total amount of arsenic found in the front half was 99 percent in each case, and at least 90 percent of this amount was found in the filter fraction. No significant amount of arsenic was present in any of the back-half components. The 0.05 mg limit reported in Table 2-2 was established after careful analysis of the sample "blank" data. These data are summarized in Section 3 of this report. In summary, 60 percent of the blank values for the NaOH rinse and H₂O impinger solutions were at or below the analytical detection limit (0.002 to 0.005 mg); the remaining blank values ranged up to a maximum value of 0.05 mg. Values below 0.05 mg were considered insignificant because the back-half arsenic content constituted less than 0.5 percent of the total arsenic collected, the liquid fraction blank data were variable, and 8.4 mg was the minimum amount of arsenic collected in any one train run for at least 60 minutes. Note that in Run

12D, the total arsenic collected was 2.7 mg. The filter frit support ruptured approximately 14 minutes into the test and sampling was immediately terminated. The train was disassembled and recovered according to routine procedures. No significant amount of arsenic was present in the back half of the train in this run, and since the concentration is comparable with the within-run data, the sample run is considered representative. Because only 0.292 dsm³ was metered, however, the sample volume does not conform to Method 108 specifications; therefore, the concentration value is not included in the group statistical data presented in Table 2-4.

The statistical data presented in Table 2-3 are comparable with data obtained during the EMSL-QAD portion of this test project. Statistical data for nine Method 108 quad train runs (36 individual samples; 121°C sample temperature) showed an overall mean of 9.59 mg/dsm³ with mean arsenic concentrations of individual quad runs ranging from 8.48 to 10.55 mg/dsm³. The standard deviations of the EMSL tests ranged from 0.10 to 1.45 mg/dsm³ with a pooled mean value of 0.59 mg/dsm³. The mean relative standard deviation (RSD) for the nine runs was 6.14 percent. Table 2-4 summarizes the EMSL quad train results.

The standard deviations of the elevated temperature quad runs ranged from 0.124 to 0.617 mg/dsm³ with a pooled mean value of 0.367 mg/dsm³. The RSD values ranged from 1.4 to 6.9 percent with a mean RSD of 4.2 percent. The mean arsenic concentration of the individual quad runs ranged from 8.20 to 9.72 mg/dsm³ with

TABLE 2-4. STATISTICAL DATA FOR GROUPED RUNS - EMSL QUAD TRAIN TESTS

	Concentration	on, ma/dsm³		
Quad run No.	Individual run value	Group _a mean	σ, mg/dsm³	RSD, ^C
1A 1B 1C 1D	9.11 8.87 6.35 9.58	8.48	1.45	17.1
2A 2B 2C 2D	8.82 10.05 9.22 9.28	9.34	0.514	5.5
3A 3B 3C 3D	9.26 10.24 10.13 10.04	9.92	0.446	4.5
4A 4B 4C 4D	9.53 9.28 8.73 9.17	9.18	0.334	3.6
5A 5B 5C 5D	10.62 10.53 10.62 10.42	10.55	0.10	0.95
6A 6B 6C 6D	9.93 9.96 9.92 10.48	10.07	0.272	2.7
7A 7B 7C 7D	9.57 9.13 9.95 9.82	9.62	0.361	3.8

(continued)

TABLE 2-4 (continued)

Quad	Concentrati	on, mg/dsm ³		
run No.	Individual run value	Group mean	σ,b mg/dsm³	RSD, ^C
8A 8B 8C 8D	10.24 9.95 9.44 9.57	9.80	0.365	3.7
9A 9B 9C 9D	9.06 9.21 9.24 9.76	9.32	0.305	3.3
Overal means	-	9.59 ^d	0.589 ^e	6.14 ^f

^aMean concentration.

epooled standard deviation;
$$\frac{\Sigma \sigma^2}{n}$$

$$f_{\overline{RSD}} = \int \frac{\Sigma \sigma^2}{n} / \bar{X}$$
.

 $^{^{\}mathrm{b}}$ Within-run standard deviation with N-1 weighting for sample data.

 $^{^{\}mbox{\scriptsize C}}\mbox{\tt Within-run}$ relative standard deviation is the standard deviation expressed as a percent of the mean concentration.

^dSimple averages of tabulate<u>d data.</u>

an overall mean of 8.74 mg/dsm³. The overall mean of the elevated temperature runs compares to within 10 percent of the overall mean of the EMSL guad runs.

As shown in Table 2-2, the Method 108 reference train tests that ran concurrently with the quad train tests are comparable relative to total arsenic concentration and distribution. Overall, there is less than a 10 percent difference between the quad run means and the reference train arsenic concentrations. In Run 10, the quad group mean was 8.89 mg/dsm³ compared with the reference train value of 8.70 mg/dsm³. In Run 11, the quad group mean excluding 11D was 8.47 mg/dsm³ and the reference train value was 9.31 mg/dsm³. The group mean in Run 12 excluding 12D was 8.68 mg/dsm³ and the reference train value was 7.78 mg/dsm³. In Run 13, the quad group mean was 8.91 mg/dsm³ compared with 9.42 mg/dsm³ for the reference train.

These data in conjunction with the EMSL quad run data suggest no significant difference between arsenic concentrations measured by Method 108 (121°C) and modified Method 108 (elevated sample temperature) at this source. The data also indicate no significant difference between samples collected at 204°C and those collected at 288°C.

Several factors observed during this test series that could have affected sample results are addressed as follows. The filter frit supports for Quad Runs 11D and 12C and 12D ruptured during testing. The ruptures were attributed to the deterioration of the silica rubber gasket due to the sample temperature

(288°C) and subsequent failure under vacuum. As mentioned in Subsection 2.2, Run 11D was not terminated when the gasket failed; consequently, a significant amount of arsenic was found in the back half of the sampling train. The arsenic measured by Train 11D, however, did not compare with the within-run samples on a total weight basis. Because particulate was noticeable on the frit, it was rinsed with 0.1 N NaOH into the container holding the contents of the first two impingers. Sample fractions from this run were reanalyzed and rechecked; no discrepancies were found in the reported analytical data.

Runs 12C and 12D were terminated immediately upon rupture of the frit. In Train 12C, the rupture occurred with less than one minute to go in the 70-minute test; in Train 12D, the rupture occurred approximately 14 minutes after the start of the test. In each case, no significant amount of arsenic was found in the back half of the train. Also, the arsenic concentrations determined in these two runs are comparable with the within-run data. New filter frits were used in each quad run, thereby minimizing this problem.

Several back-half samples (connector glassware and backup filters) from Quad Runs 10, 11, and 12 were contaminated by a brown, oily substance believed to be volatilized probe heat tape glue. This phenomenon is attributed to the high temperatures to which the probes were heated and the use of an asbestos string gasket material at the nozzle end of the probe. When heated to 288°C, the heat tape used in the construction of the probe heating system burned resulting in the volatilization of the tape

glue. A visual inspection of the affected probes showed a heavy deposit of contaminant on the asbestos gaskets as well as distinct trails of the contaminant on the nozzle end of the glass liner.

A heavier disposition of the contaminate was observed on the trains heated to 288°C than those heated to 204°C. The material was recovered by using the 0.1 N NaOH rinse and a nylon brush for each affected sample fraction. Since the material was recovered and digested according to Method 108 in a Parr bomb, any arsenic from the gas stream that might become bound to the material would be analyzed thus precluding a low bias on sample results. As the back-half results indicate (Table 2-2), no arsenic was found in these "contaminated" sample fraction; thus any bias in arsenic measurements is believed to be minimal.

Another phenomenon associated with the filter frit support occurred during the 288°C runs. Experiments conducted in our laboratory showed that the standard glass frit filter support with a silicon rubber gasket could withstand temperatures up to 260°C.

Above 260°C, deterioration of the gasket was noticeable as evidenced by a light film of material on the filter holder glassware. The material was believed to be a form of silicon oxide. This same white material was present on all of the 288°C filter glassware. A recovered sample from the laboratory experiment was analyzed for arsenic. A detection limit of 0.003 milligrams per liter was established for this sample and no detectable arsenic was found. The data indicate that this material would not cause

a high bias in arsenic results from these runs. Since the material is recoverable and would be digested in a Parr bomb, any arsenic from the gas stream which might become bound to the material would be analyzed thus precluding a low bias on sample results.

2.3 METHOD 108 TRAVERSE TEST RESULTS

Table 2-5 summarizes the sample and flue gas conditions and Table 2-6 presents the arsenic emissions data for the Method 108 traverse tests.

Triplicate tests were conducted at the completion of the quad train runs following procedures described in Method 108.

Twenty-four traverse points (12 per port) were used to traverse the cross-sectional area of the stack. Each point was sampled for 2.5 minutes yielding a total test time of 60 minutes.

Sample volumes for the three tests were consistent and ranged from 1.03 to 1.07 dsm³. Isokinetic samples rates ranged from 100.3 to 105.6 percent. The flue gas volumetric flow in actual cubic meters per minute (m³/min) averaged 886 m³/min (440 dsm³/min at 20°C and 760 mmHg). The gas temperature and moisture content averaged 254°C and 8.0 percent, respectively. Flue gas composition was determined by analyzing integrated bag samples collected during each test with an Orsat gas analyzer. Oxygen (O₂), carbon dioxide (CO₂), and carbon monoxide (CO) contents averaged 13.9, 3.8, and 0.0 percent, respectively.

As presented in Table 2-6, the total arsenic catch in milligrams ranged from 8.67 mg for Test CD-3 to 9.65 mg for Test CD-2.

TABLE 2-5. SUMMARY OF SAMPLE AND FLUE GAS CONDITIONS ARSENIC TRAVERSE TESTS

Date		Metered Moisture volume, content,		Stack gas tempera- Gas composition, a %			on,a %	Volu flow	Isoki-	
Run No.	(1984)	dsm ³	%	ture, °C	02	CO ₂	CO	m³/min	dsm³/min	netic, %
CD-1	5/19	1.03	8.0	260	14.8	3.2	0.0	851	418	105.6
CD-2	5/19	1.06	8.3	255	12.1	4.7	0.0	903	446	102.1
CD-3	5/19	1.07	7.7	248	14.8	3.5	0.0	903	455	100.3
Ave	rage	1.05	8.0	254	13.9	3.8	0.0	886	440	-

^aGas composition determined using an Orsat gas analyzer.

 $[^]b\text{Volumetric flow rate in actual cubic meters per minute (m3/min) and dry standard cubic meters per minute (dsm3/min).$

TABLE 2-6. SUMMARY OF ARSENIC ANALYTICAL RESULTS TRAVERSE TRAIN

Run No.	Metered volume, dsm³	Sample weight, mg Total arsenic	Concentra- tion, mg/dsm³	Mass emission rate, kg/h
CD-1	1.03	9.60	9.32	0.23
CD-2	1.06	9.65	9.10	0.24
CD-3	1.07	8.67	8.10	0.22
Avera	ige	9.31	8.84	0.23

Total arsenic concentration averaged 8.84 mg/dsm³ with a corresponding average mass emission rate of 0.23 kilograms per hour (kg/h). These average results obtained by multipoint, isokinetic traverse techniques are comparable to results obtained during the quad train runs.

2.4 PROCESS SAMPLES

Several finished glass samples were obtained during the test program to determine the arsenic content on a weight basis. Table 2-7 summarizes the process sample results. Results were consistent and the arsenic content by weight was approximately 0.05 percent. The samples analyzed were drinking mugs and three portions of each mug (top, handle, and bottom) were analyzed. Initially, glass chunks from the three sample fractions were placed in Teflon bombs and digested using the Parr bomb procedure from EPA Method 108. After extended heating, the glass chunks did not dissolve. Additional glass fragments were ground in an agate mortar and pestle and the resulting powder was placed in Teflon bombs and digested per the Method 108 Parr bomb procedure. After extended heating, a white precipitate remained in the bomb. The sample was filtered through a Teflon filter and the filtrate was analyzed for arsenic per Method 108. The remaining precipitate was gelatinous in nature rather than a dense powder of the original sample. The precipitate was redigested using a Parr bomb and the resulting solution was analyzed for arsenic per

TABLE 2-7. PROCESS SAMPLE ANALYTICAL RESULTS

Sample type	Lab No.	Description	Total arsenic, % by weight
Drinking mug	DM924	5/14; #2535 (handle)	0.052
	DM924	5/14; #2535 (top)	0.055
	DM924	5/14; #2535 (bottom)	0.059
Drinking mug	DM929	5/19; #2540 (handle)	0.055
	DM929	5/19; #2540 (top)	0.059
	DM929	5/19; #2540 (bottom)	0.055
	DM929R	5/19; \$2540 (bottom)	0.058

Method 108. This fraction consistently contained approximately 1 percent of the amount of arsenic found in the original filtrate. These numbers were combined and reported.

SECTION 3

PROJECT QUALITY ASSURANCE

Because the desired end product of testing is to achieve representative emission results, quality assurance is one of the main facets of stack sampling. Quality assurance guidelines provide the detailed procedures and actions necessary for defining and producing acceptable data. Five such documents were used in this test program to ensure the collection of acceptable data and to provide a definition of unacceptable data. The following documents comprise the Quality Assurance Project Plan prepared by PEI and reviewed and approved by the Environmental Monitoring Support Laboratory of the EPA (see Volume II - Appendix F); the detailed site test plan prepared by PEI and reviewed by the Emission Measurement Branch; the EPA Quality Assurance Handbook Volume III, EPA-600/4-77-027; the PEI Emission Test Quality Assurance Plan; and the PEI Laboratory Quality Assurance Plan. The last two, which are PEI's general guideline manuals, define the company's standard operating procedures and are followed by the emission testing and laboratory groups.

In this specific test program, the following steps were taken to ensure that the testing and analytical procedures produced quality data.

- ° Calibration of all field sampling equipment.
- Checks on train configuration and calculations.
- Onsite quality assurance checks (i.e., leak checks of the sampling train, pitot tube, and Orsat line) and quality assurance checks of all test equipment prior to use.
- Our Use of designated analytical equipment and sampling reagents.
- Internal and external audits to ensure accuracy in sampling and analysis.

Table 3-1 lists the sampling equipment used to perform the arsenic tests and the calibration guidelines and limits. In addition to the pre- and post-test calibrations, a field audit was performed on the metering systems and thermocouple digital indicators used in the sample runs. PEI-constructed critical orifices were used in the dry gas meter audits. The onsite audits were made at the beginning, middle, and end of the test program. Figures 3-1 through 3-23 present the results of the pre-test, mid-test, and post-test onsite audits. These data were used to assess the operational status of the sampling equipment relative to guidelines established by the U.S. EPA. The results of the three field audits indicate that the sample equipment was functioning properly throughout this test series.

Figure 3-24 is an example of an unacceptable meter box audit. The audit value for the meter coefficient deviation was greater than ±5 percent, which was considered unacceptable by the PEI Project Manager; therefore, the meter box was not used for this test program.

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TABLE 3-1. FIELD EQUIPMENT CALIBRATION Within Calibrated allowable Actual Equipment Allowable error limits ID No. against error Comments Meter box FB-1 Train A Wet test meter Y ±0.02 Y 0.004 X ΔH @ ±0.15 0.09 X (Y ±0.05 Y post-test) +1.35 χ FB-5 Train B 0.002 Χ 0.05 X Χ +0.10 FB-8 Train D 0.006 X 0.07 X -0.20 Χ FB-10 Train C 0.012 Χ 0.07 X -0.52 Χ FB-9 (Traverse 0.013 X tests) 0.08 X FB-9 (Reference +1.30 Χ train tests) Pitot tube 513 $Cp \pm 0.01$ 0K Visually Standard pitot 514 tube 0K inspected on site 508 Digital Millivolt 0.5% 0.2% 220 X indicator 221 0.4% X signals Thermocouple 411 - (stack) ASTM-3F 1.5% 0.4% Χ 412 - (stack) (±2% saturated) 0.2% X 601 - Probe 0.8% X 605 - Filter 1.0% Χ

(continued)

TABLE 3-1 (continued)

Equipment	ID No.	Calibrated against	Allowable error	Actual error	Within allowable limits	Comments
Thermocouple (cont'd)	614 - Probe 615 - Probe			0.4% 0.6%	X	
	616 - Probe 618 - Probe			0.5% 0.6%	X X	
	619 - Probe 620 - Filter			0.6% 1.2%	X X	
Orsat analyzer	141	Standard gas	±0.5%	0.0% 0.2% 0.0%	X X X	c6 c6
Impinger thermometer	433 434 435 446 385	ASTM-3F	±2°F	1.2°F 0.5°F 1.0°F 1.0°F	X X X X	
Mettler balance	M-1	Type S weights	±0.5 g	+0.1 g	Х	
Barometer	407	NBS traceable barometer	+0.10 in.Hg. (0.20 post-test)	0.01 in.Hg.	х	
Dry gas thermometer	FB-1	ASTM-3F	±5°F	4°F 5°F	X	Inlet Outlet
	FB-5			4°F 2°F	X	Inlet Outlet
	FB-8			2°F 3°F	X	Inlet Outlet

(continued)

TABLE 3-1 (continued)

Equipment	ID No.	Calibrated against	Allowable error	Actual error	Within allowable limits	Comments
Dry gas thermometer (cont'd)	FB-10 FB-9			2°F 2°F 2°F	X X	Inlet Outlet Inlet
	10-5			2°F	X X	Outlet
Probe nozzle	1A 1B 1C 1D 2A 2B 2C 2D 5-106 (RT tests) 2-117 (Traverse tests)	Caliper	Dn ±0.004 in.	0.001 in. 0.002 in. 0.001 in. 0.002 in. 0.002 in. 0.002 in. 0.002 in. 0.001 in.	x X X X X X X X X X X X X	

DATE: 5-11-84

BAROMETRIC PRESSURE (Pbar): 29.00 in. Hg

ORIFICE NO. 6

ORIFICE K FACTOR: 5.241110-4

ORIFICE K FACTOR: 5.241110-4

ORIFICE K FACTOR: 5.241110-4

ORIFICE K FACTOR: 5.241110-4

Orifice	Dry gas		emperatures			Duration	
manometer	meter	Ambient		Dr			of
reading ΔH,	reading V _i /V _f ,	T _{ai} /T _{af} ,	Average T _a ,	Inlet T _{ii} /T _{if} ,	Outlet T _{oi} /T _{of} ,	Average T _m ,	run Ø min.
in.H ₂ 0	ft³	°F	°F	°F	°F	°F	
	894.500	68		72	64		,7/23
2.05	909,200	70	69	72	65	68.25	1760

Dry gas meter V _m , ft ³	V mstd' ft ³	V mact' ft³	Audit,	Y devia- tion,%	Audit ^{AH0} , in.H ₂ 0	∆H@ Devia- tion, in.H ₂ O
14.700	14.315	13.673	0.955	1.05	1,72	0.0

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = /4.315 \text{ ft}^{3}$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = /3.673 \text{ ft}^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = 0.955$$
 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = 1.05$

Audit
$$\triangle H0 = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)} \right]^2 = / \frac{2}{12} in.H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\Delta H0$ must be in the range pre-test $\Delta H0$ ± 0.15 inches H_2O .

Figure 3-1. Pre-test audit report: dry gas meter by critical orifice (Meter Box FB-1, Train A).

DATE: 5-11-84

BAROMETRIC PRESSURE (Pbar): 29.00 in. Hg

ORIFICE NO. 3

ORIFICE K FACTOR: 5,377 × 10-9

AUDITOR: barole Figural Control of the control of th

Orifice	Dry gas		Ţ	emperatures			Duration	
manometer	meter	Ambient		Dr		of		
reading ΔH ,	reading V _i /V _f ,	Tai/Taf'	Average T _a , °F	Inlet T _{ij} /T _{if} ,	Outlet T _{oi} /T _{of} ,	Average T _m ,	run Ø min.	
in.H ₂ 0	948.70c		F	79	7	<u> </u>		
2.25	263,60		70	80	76	77.5	1732	

Dry gas meter V _m , ft ³	V mstd' ft ³	V mact' ft³	Audit, Y	Y devia- tion, %	Audit ΔH@, in.H _z O	ΔH@ Devia- tion, in H ₂ O
14.900	14.267	13,892	0.974	0.92	1,77	0,07

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = /4.26 7 ft^{3}$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = /3.892 \text{ ft}^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = 0.974$$
 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -0.92$

Audit
$$\triangle H0 = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)}\right]^2 = /.77 \text{ in.} H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\Delta H0$ must be in the range pre-test $\Delta H0$ ± 0.15 inches H₂O.

Figure 3-2. Pre-test audit report: dry gas meter by critical orifice (Meter Box FB-5, Train B).

DATE: 5-11-84

BAROMETRIC PRESSURE (Pbar): 39.00 in. Hg

ORIFICE NO. 3

ORIFICE K FACTOR: 5.327 ×10-4

ORIFICE K FACTOR: 5.327 ×10-4

CLIENT: USCPA-QAD

METER BOX NO. FB 8 - TRAID

PRETEST Y: 0.990 AHD 191 in. H₂C

AUDITOR: Janiel Statement

Orifice	Dry gas		T	emperatures			Duration
manometer reading ΔH , in.H ₂ O	nometer meter reading V _i /V _f ,	Ambi T _{ai} /T _{af} ,	Average T _a ,	Inlet T _{ii} /T _{if} , °F	y gas meter Outlet Toi ^{/T} of, °F	Average T _m , °F	of run Ø min.
2.30	414.300	72	72	73 74	69	71.5	15 26.2

Dry gas meter V _m , ft³	V mstd' ft ³	V mact' ft³	Audit, Y	Y devia- tion, %	Audit ΔH0, in.H ₂ O	ΔH@ Devia- tion, in H ₂ O
13.200	12.784	12:577	0.982	0.81	1.84	0.07

$$V_{\text{mstd}} = \frac{17.647(V_{\text{m}})(P_{\text{bar}} + \Delta H/13.6)}{(T_{\text{m}} + 460)} = 12.784 \text{ ft}^3$$

$$V_{\text{mact}} = \frac{1203(\%)(K)(P_{\text{bar}})}{(T_a + 460)} = /2.557 \text{ft}^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = 0.982$$
 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = 0.81$

Audit
$$\triangle H0 = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)} \right]^2 = /94 \text{ in.} H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\Delta H0$ must be in the range pre-test $\Delta H0$ ± 0.15 inches H₂O.

Figure 3-3. Pre-test audit report: dry gas meter by critical orifice (Meter Box FB-8, Train D).

DATE: 5-11-84

BAROMETRIC PRESSURE (P_{bar}): 29.00 in. Hg

ORIFICE NO. /2

ORIFICE K FACTOR: 4.795 ×10-4

Orifice Dry gas Temperatures

manometer meter Ambient Dry gas meter of reading reading To // To Javerage Inlet Outlet Average run

Orifice	Dry gas		Duration				
manometer	meter	Ambient		Dr		of	
reading ΔH, in.H ₂ O	reading V _i /V _f ,	Tai ^{/T} af'	Average T _a , °F	Inlet T _{ii} /T _{if} , °F	Outlet T _{oi} /T _{of} , °F	Average T _m , °F	run Ø min.
	020,900	70		76	72		A.O
1.80	037,900	71	70.5	77	74	74,75	2160

Dry gas meter V _m , ft³	V _m std' ft ³	V mact, ft ³	Audit,	Y devia- tion, %	Audit ΔH0, in.H ₂ O	ΔH@ Devia- tion, in.H ₂ O
17.000	16.343	15,484	0.947	-2.1	1,75	0.06

$$v_{\text{mstd}} = \frac{17.647(v_{\text{m}})(P_{\text{bar}} + \Delta H/13.6)}{(T_{\text{m}} + 460)} = 16.343 \text{ft}^3$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = 15.484 \text{ft}^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = \frac{947}{947}$$
 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -2.1$

Audit
$$\triangle H0 = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)} \right]^2 = 1.75 \text{ in.} H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\Delta H0$ must be in the range pre-test $\Delta H0$ ± 0.15 inches H₂O.

Figure 3-4. Pre-test audit report: dry gas meter by critical orifice (Meter Box FB-10, Train C).

DATE: 5-16-84

CLIENT: USEPA
BAROMETRIC PRESSURE (Pbar): 29.52 in. Hg

ORIFICE NO. #7

ORIFICE K FACTOR: 4.964410-4

AUDITOR: Namel Follows

Orifice manometer	Dry gas meter	Ambi		<u>emperatures</u> Dr	y gas meter		Duration of
reading ΔH,	reading V _i /V _f ,	T _{ai} /T _{af} ,	Average T _a ,	Inlet T _{ii} /T _{if} ,	Outlet Toi/Tof,	Average T _m ,	run Ø min.
in.H ₂ 0	ft³	°F	°F	°F	°F	°F	
_	073.000	72	7- 0	82	72	-0-	1.9
2,25	043,200	7480	73.0	84	76	78.5	2560

Dry gas meter V _m , ft ³	V mstd' ft³	V mact' ft³	Audit,	Y devia- tion, %	Audit ∆H0, in.H ₂ O	ΔH@ Devia- tion, in.H ₂ O
20.2	20.123	19.112	0.950	-3.15	2.05	0.02

$$V_{\text{m}_{\text{std}}} = \frac{17.647(V_{\text{m}})(P_{\text{bar}} + \Delta H/13.6)}{(T_{\text{m}} + 460)} = 20.123\text{ft}^3$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = /9.1/2 \text{ ft}^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = 0.950 \text{ Y deviation} = \frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -3.75$$

Audit
$$\triangle H@ = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)}\right]^2 = 2.05 \text{ in.} H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\triangle H0$ must be in the range pre-test $\triangle H0$ ±0.15 inches H₂O.

> Figure 3-5. Pre-test audit report: dry gas meter by critical orifice (Meter Box FB-9, Single Point-Traverse Tests).

Date <u>5-1/</u>	<u>- 84</u> in	dicator No	Operator _	Vef
Test Point No.	Millivolt signal*	Equivalent temperature,	Digital indicator temperature reading, *F	Difference,
1		32	32	0.0
2		200	202	- 0.30
3		540	540	0.0
4		1194	1196	-0.12

Percent difference must be less than or equal to 0.5%.

Percent difference:

(Equivalent temperature °R - Digital indicator temperature reading °R)(100%)
(Equivalent temperature °R)

Where $^{\circ}R = ^{\circ}F + 460^{\circ}F$

Figure 3-6. Pre-test thermocouple digital indicator audit data sheet (Indicator No. 220).

These values are to be obtained from the calibration data sheet for the calibration device.

Date 5-11-84 Indicator No. 221 Operator Digital indicator Equivalent Difference. Test Point Millivolt temperature, temperature reading. of* signal* % No. 32 0.41 1 -0.15 2 201 200 3 0.00 一0.12 4

Percent difference must be less than or equal to 0.5%.

Percent difference:

(Equivalent temperature °R - Digital indicator temperature reading °R)(100%)
(Equivalent temperature °R)

Where $^{\circ}R = ^{\circ}F + 460^{\circ}F$

Figure 3-7. Pre-test thermocouple digital indicator audit data sheet (Indicator No. 221).

These values are to be obtained from the calibration data sheet for the calibration device.

ON-SITE AUDIT DATA SHEET

	Equipment	Refer	ence	Reference Value	Determi		Devia	tion		Allowable viation
2 1 5	Meter box inlet thermo.	ASTM- ambie	3F at nt temp.	68 68	71 66 72		3 2 4			5°F
Z	Meter box outlet thermo.	ASTM- ambie	3F at nt temp.	68 68	71 62 70		34			5°F
16	Impinger thermometer		3F at nt temp.	66	64		2			2°F
5·	thermometer ambig		3F at nt temp.	70	68 68		Z Z			7°F
0 -0	Thermocouple	ASTM-3F at stack temp.		70 70	67		3 3		Sa	e table
•	Orsat analyzer	% O ₂ ambie	in nt air	20.8%			-			0.7%
	Trip balance	IOLM weigh		NA	NA		N	A	0.	5 grams
	Barometer	Corre NWS v	cted* alue						0.2	20 in. Hg
:	Reference temp	. °F	32-140	141-273	274-406	407-	-540	541-	673	674-760
•	Max. deviation	°F	7	9	11		13	1	5	17

* Correction factor:

NWS value (in. Hg) - [Altitude (ft)/1000(ft/in. Hg)] + 0.74 in. Hg**

If it is not feasible to perform the audit on any piece of equipment, record "N/A" in the space provided for the data.

Figure 3-8. Pre-test onsite audit data sheet.

^{** 0.74} in. Hg is the nominal correction factor for the reference barometer against which the field barometer was calibrated.

ON-SITE AUDIT DATA SHEET

	Audit Name	NOI	ANA G	US	Date: <u>5-/</u>	//-8	4 1	ludito	r	
	Equipment	Refer	ence	Reference Value	Value Determi	-	Devia	ition		Allowable viation
FB 8	Meter box inlet thermo.		3F at nt temp.	68	67 65		3			5°F
	Meter box outlet thermo.		3F at	68	66	,	2			5°F
433 435	Impinger thermometer			66	66			0		2°F
••	Stack thermometer		3F at ent temp.							7°F
	or Thermocouple	1	3F at temp.						Se	e table
	Orsat analyzer	% O ₂ ambie	in ent air	20.8%						0.7%
	Trip balance	IOLM weigh							0.	5 grams
	Barometer	Corre	ected* value						0.2	20 in. Hg
	Reference temp	· °F	32-140	141-273	274-406	407	-540	541-	673	674-760
	Max. deviation	°F	7	9	11		13	1	5	17

* Correction factor:

NWS value (in. Hg) - [Altitude (ft)/1000(ft/in. Hg)] + 0.74 in. Hg**

If it is not feasible to perform the audit on any piece of equipment, record "N/A" in the space provided for the data.

Figure 3-9. Pre-test onsite audit data sheet.

^{** 0.74} in. Hg is the nominal correction factor for the reference barometer against which the field barometer was calibrated.

DATE: 5-16-84

BAROMETRIC PRESSURE (Pbar): 29.52 in. Hg

ORIFICE NO. #3

ORIFICE K FACTOR: 5.379×10⁻⁴

AUDITOR: Manual Factor: 5.379×10⁻⁴

ORIFICE K FACTOR: 5.379×10⁻⁴

AUDITOR: Manual Factor: 5.379×10⁻⁴

Orifice	Dry gas		T	emperatures			Duration of run Ø min.
manometer reading ΔH, in.H ₂ O		Ambi T _{ai} /T _{af} ,	Average T _a ,	Dr Inlet T _{ii} /T _{if} ,	y gas meter Outlet T _{oi} /T _{of} ,	Average T _m ,	
	261.900	69	,	\$3	78	r	54.
2,20	276.200		69	80	77	79.5	1560

Dry gas meter V _m , ft ³	V mstd' ft ³	V mact' ft³	Audit, Y	Y devia- tion, %	Audit ∆H0, in.H ₂ O	ΔH@ Devia- tion, in.H ₂ O
14.300	13.884	/3.700	0.951	-1.47	1.67	0.05

$$V_{\text{m}_{\text{std}}} = \frac{17.647(V_{\text{m}})(P_{\text{bar}} + \Delta H/13.6)}{(T_{\text{m}} + 460)} = /3.884 \text{ ft}^3$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = /3.200 \text{ ft}^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = 0.951$$
 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -1.47$

Audit
$$\triangle H0 = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)}\right]^2 = /.67 \text{ in.H}_20$$

Audit Y must be in the range, pre-test Y ±0.05 Y. Audit $\Delta H0$ must be in the range pre-test $\Delta H0$ ±0.15 inches H₂0.

> Figure 3-10. Mid-test audit report: dry gas meter by critical orifice (Meter Box FB-1).

DATE: 5-16-84 CLIENT: USEPA-QAN

BAROMETRIC PRESSURE (Pbar): 2952 in. Hg

ORIFICE NO. 7

ORIFICE K FACTOR: 4.964×10-4

AUDITOR: 15-16-84

CLIENT: USEPA-QAN

METER BOX NO. FB#5

PRETEST Y: 6.983 AHP 1.84 in. H₂0

AUDITOR: 15-16-84

Orifice	Dry gas meter reading V _i /V _f , ft ³		T	e mperatures			Duration of run Ø min.
manometer reading ΔH, in.H ₂ O		Ambi T _{ai} /T _{af} ,	ent Average T _a , °F	Inlet T _{ii} /T _{if} , °F	y gas meter Outlet Toi ^{/T} of, °F	Average T _m , °F	
2.10	638.500 652.500		70	98	92	93.75	10 60

Dry gas meter V _m , ft ³	V mstd' ft ³	V mact' ft³	Audit,	Y devia- tion, %	Audit ∆H@, in.H ₂ O	ΔH@ Devia- tion, in.H ₂ O
14.000	13.239	12.795	0.966	-1.76	1.82	0.02

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = /3.239 \text{ ft}^{3}$$

$$V_{m_{act}} = \frac{1203(0)(K)(P_{bar})}{(T_a + 460)} = /2.795 ft^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = 0.966$$
 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -1.76$

Audit
$$\triangle H0 = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)} \right]^2 = /.8 \times in.H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\Delta H0$ must be in the range pre-test $\Delta H0$ ± 0.15 inches H₂O.

Figure 3-11. Mid-test audit report: dry gas meter by critical orifice (Meter Box FB-5).

BAROMETRIC ORIFICE NO	5-/6- C PRESSURE (D. 76 FACTOR: 2	(P _{bar}): <u>29</u>		CLIENT: USEFA-QAD METER BOX NO. FB+8 PRETEST Y-0.990 AND (9/ 1n.H. AUDITOR: Variof- Filterer				
Orifice manometer reading ΔH , in. H_2O	Dry gas meter reading V ₁ /V _f , ft ³	Ambi T _{ai} /T _{af} ,		emperatures Dr Inlet T _{1i} /T _{if} , °F	y gas meter Outlet T _{oi} /T _{of} , °F	Average T _m ,	Duration of run Ø min.	
2.25	797.000 790.500	69 70	69.5	88	36 37	87.25	1560	

Dry gas meter V _m , ft ³	V mstd' ft ³	V mact' ft³	Audit,	Y devia- tion, %	Audit ∆H0, in.H₂O	ΔH@ Devia- tion, in.H ₂ O
13 500	12923	12.747	0986	0,40	1.82	0.09

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = 12.923 \text{ ft}^{3}$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = /2.747_{ft^3}$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = 0.986$$
 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -0.40$

Audit
$$\triangle H0 = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)} \right]^2 = /.82 \text{ in.} H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit Δ H0 must be in the range pre-test Δ H0 ± 0.15 inches H20.

Figure 3-12. Mid-test audit report: dry gas meter by critical orifice (Meter Box FB-8).

DATE: 5-16-84 CLIENT: USEPA-QADBAROMETRIC PRESSURE $(P_{bar}): 2952$ in. Hg METER BOX NO. FB# 10ORIFICE NO. \$412 PRETEST Y: 9967 \$49 \$81 in. H₂0

AUDITOR: ORIFICE K FACTOR: Temperatures Duration Orifice Dry gas Ambient Dry gas meter manometerl meter of reading Average Inlet Outlet Average run ΔН, Ø

reading V_1/V_f , V_1/V_f , V

Dry gas meter V _m , ft ³	V mstd' ft ³	V mact' ft³	Audit,	Y devia- tion, %	Audit ΔH0, in.H ₂ O	ΔH@ Devia- tion, in.H ₂ O
12.700	12.053	11.442	0.949	-1.90	1.77	0.04

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = /2.053 \text{ ft}^{3}$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = //.44Z_{\text{ft}^3}$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = 0.949$$
 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -1.90$

Audit
$$\triangle H@ = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)}\right]^2 = 1.77 \text{ in.} H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\Delta H0$ must be in the range pre-test $\Delta H0$ ± 0.15 inches H₂O.

Figure 3-13. Mid-test audit report: dry gas meter by critical orifice (Meter Box FB-10).

Date <u>5-/</u>	<u>6-84</u> in	dicator No	0perator	Vol.
Test Point No.			Digital indicator temperature reading, *F	Difference,
1		32	33	2
2		200	203	, ,45
3		540	539	.1
4		1194	1194	0

Percent difference must be less than or equal to 0.5%.

Percent difference:

Where $^{\circ}R = ^{\circ}F + 460^{\circ}F$

Figure 3-14. Mid-test thermocouple digital indicator audit data sheet (Indicator No. 220).

These values are to be obtained from the calibration data sheet for the calibration device.

Date <u>5-1</u>	Date 5-16-84 Indicator No. 22/ Operator 12/									
Test Point No.	Millivolt signal*	Equivalent temperature,	Digital indicator temperature reading, *F	Difference,						
1		32	29	r.61						
2		200	199	.16						
3		540	537	.3⁄						
4		1194	1193	.06						

Percent difference must be less than or equal to 0.5%.

Percent difference:

Where $^{\circ}R = ^{\circ}F + 460^{\circ}F$

Figure 3-15. Mid-test thermocouple digital indicator audit data sheet (Indicator No. 221).

These values are to be obtained from the calibration data sheet for the calibration device.

ON-SITE AUDIT DATA SHEET

	Equipment	Reference	Reference Value	Value Determined	Deviation	Max. Allowable Deviation
3 K 35 V	Meter box inlet thermo.	ASTM-3F at ambient temp.	58 58	57 56	2	5°F
BIC	Meter box outlet thermo.	ASTM-3F at ambient temp.	58 58	55 55	3	5°F
133 1 34	Impinger thermometer	ASTM-3F at ambient temp.	64 64	64	0 2	2°F
· · ·	Stack thermometer	ASTM-3F at ambient temp.				7°F
	Thermocouple	ASTM-3F at stack temp.				See table
	Orsat analyzer	% 0 ₂ in ambient air	20.8%			0.7%
	Trip balance	IOLM std. weight	NA			0.5 grams
	Barometer	Corrected* NWS value	NB			0.20 in. Hg

Reference temp. °F	32-140	141-273	274-406	407-540	541-673	674-760
Max. deviation °F	7	9	11	13	15	17

* Correction factor:

NWS value (in. Hg) - [Altitude (ft)/1000(ft/in. Hg)] + 0.74 in. Hg**

If it is not feasible to perform the audit on any piece of equipment, record "N/A" in the space provided for the data.

Figure 3-16. Mid-test onsite audit data sheet.

^{** 0.74} in. Hg is the nominal correction factor for the reference barometer against which the field barometer was calibrated.

ON-SITE AUDIT DATA SHEET

	Equipment	Reference	Reference Value	Value Determine	d Devia		Allowable viation
9	Meter box inlet thermo. Meter box outlet thermo.	ASTM-3F at ambient temp.	58 58	57 54 59	-1		5°F
59° 51 35	Meter box outlet thermo.	ASTM-3F at ambient temp.	58 58 58	53 53 58	W 0		5°F
6	Impinger thermometer	ASTM-3F at ambient temp.	64	66	1 2		2°F
	Stack thermometer	ASTM-3F at ambient temp.					7°F
	or Thermocouple	ASTM-3F at stack temp.				Se	ee table
	Orsat analyzer	% 0 ₂ in ambient air	20.8%	·			0.7%
	Trip balance	IOLM std. weight	AN			0	.5 grams
:	Barometer	Corrected* NWS value	Ah			0.	20 in. Hg
:	Reference temp	. °F 32-140	141-273	274-406	407-540	541-673	674-760
•	May dayintia	or 7				1.5	1.7

17 11 13 15

* Correction factor:

NWS value (in. Hg) - [Altitude (ft)/1000(ft/in. Hg)] + 0.74 in. Hg**

If it is not feasible to perform the audit on any piece of equipment, record "N/A" in the space provided for the data.

Figure 3-17. Mid-test onsite audit data sheet.

^{** 0.74} in. Hg is the nominal correction factor for the reference barometer against which the field barometer was calibrated.

DATE: 5/19/84 CLIENT: USEPA

BAROMETRIC PRESSURE (Pbar): 29.10 in. Hg

ORIFICE NO. 3

ORIFICE K FACTOR: 5.377×10-4

AUDITOR: Phillips

Orifice	Dry gas		T	emperatures			Duration	
manometer reading	meter reading	Ambient T _{ai} /T _{af} , Average		Dry gas meter Inlet Outlet		Average	of run	
ΔΗ, in.H ₂ O	V ₁ /V _f , ft³	۰۴	°F	T _{ii} /T _{if} ,	T _{oi} /T _{of} ,	m* °F	min.	
2.15	490,025	78	78	79	71	76.25	15.0	
2.10	5 03,330	78	18	82	73	16.23	73.0	

Dry gas meter V _m , ft ³	V _m std, ft ³	V mact' ft³	Audit, Y	Y devia- tion, %	Audit ΔH0, in.H ₂ O	ΔH@ Devia- tion, in H ₂ O
13.305	12,810	12.173	.950	-1.5P	1.75	+0.03

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = 12.710 \text{ ft}^{3}$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = /2.173 \text{ ft}^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}}$$
 = .450 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}}$ x 100 = -/.572

Audit
$$\triangle H@ = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)}\right]^2 = 1.75 \text{ in.} H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\Delta H0$ must be in the range pre-test $\Delta H0$ ± 0.15 inches H₂O.

Figure 3-18. Post-test audit report: dry gas meter by critical orifice (Meter Box FB-1).

DATE: 5/19/84

BAROMETRIC PRESSURE (Pbar): 29.10 in. Hg

ORIFICE NO. 12

ORIFICE K FACTOR: 4.795x 10-4

CLIENT: USEPA

METER BOX NO. FB-5

PRETEST Y: 0.983

AUDITOR: Philips

Orifice	Dry gas			Duration of			
manometer		Ambient			Dr		
reading ΔH,	reading V _i /V _f ,	T _{ai} /T _{af} ,	Average T _a ,	Inlet T _{ii} /T _{if} ,	Outlet T _{oi} /T _{of} ,	Average T _m ,	run Ø min.
in.H ₂ O	ft³	°F	°F	°F	°F	°F	
1.90	884.730	78	70	86	80	QV	150
1.10	896.520	78	78	88	82	• 7	15.0

Dry gas meter V _m , ft ³	V _m std' ft³	V mact' ft ³	Audit, Y	Υ devia- tion, %	Audit ΔH0, in.H ₂ O	$\Delta H0$ Devia- tion, in H_20
11.790	11.183	10.855	1971	-1.24	1.91	+,07

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = 11.163 \text{ ft}^{3}$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = /0.855 \text{ ft}^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = 97$$
/ Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -1.24\%$

Audit
$$\triangle H@ = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)}\right]^2 = /.9/ in.H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit Δ H0 must be in the range pre-test Δ H0 ± 0.15 inches H20.

Figure 3-19. Post-test audit report: dry gas meter by critical orifice (Meter Box FB-5).

DATE: 5/19/84 CLIENT: USEPA

BAROMETRIC PRESSURE (Pbar): 21/0 in. Hg METER BOX NO. FB-8

ORIFICE NO. 7 PRETEST Y: 0.990 AHO 1.91 In.H20
ORIFICE K FACTOR: 4.964×10-4 AUDITOR: Phillips

Orifice	Dry gas			Duration			
manometer	meter	Ambient		Dr	of		
reading ΔH ,	reading V ₁ /V _f ,	Tai/Taf,	Average T _a ,	Inlet T _{ii} /T _{if} ,	Outlet T _{oi} /T _{of} ,	Average T _m ,	run Ø min.
in.H ₂ O	ft³	۰F	°F	°F	°F	°F	
	017500	78	P0	84	20	P3.5	39-3
2.10	049.200	82		87	33		5/6

Dry gas meter V _m , ft ³	V mstd' ft ³	V mact' ft³	Audit, Y	Y devia- tion, %	Audit ΔH0, in.H ₂ O	ΔH@ Devia- tion, in H ₂ O
31.400	29.824	29.257	.991	92	1.98	+,07

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = 29.P2L ft^{3}$$

$$V_{\text{mact}} = \frac{1203(0)(K)(P_{\text{bar}})}{(T_a + 460)} = 29.257 \text{ft}^3$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = .9f$$
 / Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -.92\%$

Audit
$$\triangle H@ = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)}\right]^2 = 1.9$$
 in.H₂0

Audit Y must be in the range, pre-test Y ±0.05 Y. Audit AH@ must be in the range pre-test AH@ ±0.15 inches H₂O.

> Figure 3-20. Post-test audit report: dry gas meter by critical orifice (Meter Box FB-8).

	5/19/84	0 1. 2	? (D in Un	CLIENT: USEPA METER BOX NO. FB-10 PRETEST Y:0,967 AHO [.8] In.H. AUDITOR: Phillips			
ORIFICE NO		0	*F				
Orifice manometer	Dry gas meter	Ambi		emperatures Dr			Duration of
reading ΔH,	reading V ₁ /V _f ,	Tai Taf'	Average T _a ,	Inlet T _{ii} /T _{if} ,	Outlet	Average T _m ,	run Ø min.
in.H ₂ 0	ft³	°F	°F	°F	۰Ł	°F	111111
	222 800	78		94	20	00	103

Dry gas meter V _m , ft ³	V mstd' ft ³	V mact, ft ³	Audit, Y	Y devia- tion, %	Audit ∆H@, in.H ₂ O	ΔH@ Devia- tion, in H₂O
17,100	16.128	15,5P7	.966	-,10	1.85	+,04.

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = 16.12 \Gamma_{ft^{3}}$$

$$V_{\text{mact}} = \frac{1203(P)(K)(P_{\text{bar}})}{(T_a + 460)} = /5.5f^{3}$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}} = .966$$
 Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = -.102$

Audit
$$\Delta HC = (0.0317)(\Delta H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \Delta H/13.6)}\right]^2 = 1.65 \text{ in.} H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\Delta H0$ must be in the range pre-test $\Delta H0$ ± 0.15 inches H₂O.

Figure 3-21. Post-test audit report: dry gas meter by critical orifice (Meter Box FB-10).

Date 5/19/84 Indicator No. 220 Operator Phillips									
Test Point No.	Millivolt signal*	Equivalent temperature,	Digital indicator temperature reading,	Difference,					
1		32	33	.20					
2		200	202	,30					

,or

,00

540

Percent difference must be less than or equal to 0.5%.

Percent difference:

3

(Equivalent temperature °R - Digital indicator temperature reading °R)(100%)
(Equivalent temperature °R)

Where $^{\circ}R = ^{\circ}F + 460^{\circ}F$

Figure 3-22. Post-test thermocouple digital indicator audit data sheet (Indicator No. 220).

These values are to be obtained from the calibration data sheet for the calibration device.

Date 5-19-84 Indicator No. 221 Operator bush growth

Test Point No.	Millivolt signal*	Equivalent temperature,	Digital indicator temperature reading, *F	Difference,
1		32	29	.61
2		200	199	.15
3		540	5 38	.17
4		1194	1194	,00

Percent difference must be less than or equal to 0.5%.

Percent difference:

(Equivalent temperature *R - Digital indicator temperature reading *R)(100%)
(Equivalent temperature *R)

Where R = F + 460°F

These values are to be obtained from the calibration data sheet for the calibration device.

Figure 3-23. Post-test thermocouple digital indicator audit data sheet (Indicator No. 221).

BAROMETRIC ORIFICE NO	-//-84 C PRESSURE (D. <u>7</u> FACTOR: _4			METER BOX	0.989	<u>ع</u>	3 1n.H ₂ 0
Orifice	Dry gas			emperatures			Duration
manometer reading	meter reading	Ambient T_/T_, Average		Dry gas meter Inlet Outlet Avera			of run
ΔH,	V ₄ /V _f ,	ai/laf'	T _a ,	Tii/Tif,	Toi/Tof,	T _m ,	. 0
in.H ₂ 0	ft³	°F	°F	°F	°F	°F	min.
	624.800	68		70	67		1, 18.2
1.90	624.800 638.300	68	68	70	67	68.5	1660
		 					

Dry gas meter V _m , ft³	V mstd' ft ³	V mact' ft³	Audit,	Y devia- tion, %	Audit ∆H@, in.H ₂ O	ΔH@ Devia- tion, in H ₂ O
13.500	13.138	12.285	0.935	5.78	1.62	0.10 V

$$V_{m_{std}} = \frac{17.647(V_{m})(P_{bar} + \Delta H/13.6)}{(T_{m} + 460)} = /3./36 ft^{3}$$

$$V_{m_{act}} = \frac{1203(0)(K)(P_{bar})}{(T_{a} + 460)} = /3.285 ft^{3}$$

Audit Y =
$$\frac{V_{\text{mact}}}{V_{\text{mstd}}}$$
 = Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = 5.78$

Audit
$$\triangle H0 = (0.0317)(\triangle H)(P_{bar})(T_m + 460) \left[\frac{\emptyset}{Y(V_m)(P_{bar} + \triangle H/13.6)}\right]^2 = 1.62in.H_20$$

Audit Y must be in the range, pre-test Y ± 0.05 Y. Audit $\Delta H@$ must be in the range pre-test $\Delta H@$ ± 0.15 inches H_2O .

Figure 3-24. Example of unacceptable dry gas meter audit.

PEI personnel calculated the sampling rates on site. The data were rechecked and validated at the end of the test program by computer programming. Some minor discrepancies between the hand calculations and computer printouts resulted primarily because of round-off error. Overall, the data compared favorably. Figure 3-25 presents an example calculation form PEI using during this test program. Computerized example calculations are presented in Appendix A.

As an additional check of the reliability of the method used to analyze the samples, two blank trains were assembled in the recovery area, capped off, and set aside for about 2 hours. first blank train was assembled at the beginning of the test series using clean glassware. On the same day as Quad Runs 7, 8, and 9 (EMSL work), the second blank train was assembled with glassware used during previous sampling runs. The blank trains were recovered in the same manner as the test samples. These samples were shipped to the laboratory and analyzed by the same procedures as those used for the actual emission samples. addition to the blank sampling train, aliquots of the field reagents used in the collection and recovery of the samples were obtained daily and analyzed by the same procedures as those used for the actual samples. Table 3-2 presents the results of the blank sample trains and field blank analyses. The results are considered reasonable and indicate that background arsenic contamination was not a problem in the sample recovery area. The results of the blanks are relatively small.

51	TE Indiana Glass	lo,	_ TEST NO			
_			RUN 1	RUN 2	RUN 3	A C
1.	Volume of dry gas sampled corrected to standard conditions. Note: V must be corrected for leakage if any likekage	Y _m , ft ³	39.977	41.533	44.003	52.02
	corrected for leekage if any leekage rates gareed La).	٧	.965	. 983	.967	.980
	V _m = 17.65 x V _m x V P _{ber + 17.6} .	P _{bar} , in.Hg	29.48	29.48	29.48	29.48
		ĀĦ, 1n.H ₂ 0	.903	1.16	1.15	2.1!
	13,226 40)	Ť _m , ◆R	529	544	534	53!
	57.1° ⁷	V _m , dscf	38,024	39.156	41.573	52.74
2.	Volume of water vapor at standard con- ditions, ft.	ν _{lc} , g	73.8	86.4	81.4	93.4
	V = 0.04707V _{1c} -	V _{wstd} ,ft ³	3.47	4.07	3.83	4-40
3.	Moisture content in stack gas.	Bws	.064	,094	.084	.077
	Bus " Wetd " " " " " " " " " " " " " " " " " " "	1-B _{ws}	.916	.906	.916	,923
4.	Dry molecular weight of stack gas, 1b/1b-mole.	% CO ₂	4.3			
	Mg = 0.440 (\$ CO2) + 0.320 (\$ O2)	* 502 02	רגו		-	
	+ 0.280 (\$ R ₂ + \$ CO) +	1 N ₂ + 1 CO	83			-
		M _d , lb/lb-mole	29.2			
5.	Molecular weight of stack gas. M _S = M _d (1-B _{WS}) + 18 B _{WS} =	M _s , 1b/1b-mole	28,259			28.3
6.	fps	Pstatic, in.H20	53			>
	V _g = 85.49 Cp (avg. √aF) √ T _g =	P _s , in.Hg	29,44			>
	"s "s	T _s , *R	103E			>/0
		√ AP	.540			.598
		Ср	.84	1		
		V _s , fps	43.3			41.8
7.	Isotinetic variation	Dn, in.	,287			.3/9
	8 1 + Vasu x Ts x 17.32	e, min.				
	ν _s = 0 _n ² = 0 = P _s = (1-0 _{es})	S 1	101.3			102.0

Figure 3-25. Example of onsite calibration data sheet.

TABLE 3-2. ARSENIC BLANK DATA

Blank sample train arsenic values ^a						
Train No.	Filter, µg	NaOH probe rinse, μg	Impinger section, μg	Total train blank, µg		
1	32.8	8.0	22.2	63.0		
2	30.8	7.8	11.6	50.2		

Field blank arsenic values

Date samples taken	Corresponding Run No.	Filter total, µg	NaOH ^b , mg/liter	H ₂ O ^C , mg/liter
5/17/84	10 + 11	26.7	0.0168	0.0085
5/18/84	12 + 13	29.3	0.0137	0.0101
5/19/84	CD1 - CD3	28.8	0.0153	0.0111
Averag	Average blank values		0.0153	0.0099

^aSample train was fully assembled in recovery area and then recovered and analyzed as a sample.

^bBetween 235 and 238 ml of NaOH was used to rinse the probe. Between 179 and 456 ml of the NaOH was used to rinse Impingers 1 and 2. Between 128 and 184 ml of the NaOH was used to rinse Impingers 3 and 4. Between 119 and 180 ml of the NaOH was used to rinse the connector. The maximum blank for the NaOH corresponds to 6 μ g for the probe rinse, 8 μ g for the impinger samples, and 3 μ g for the connector samples.

 $^{^{\}text{C}}\text{On}$ both days, 300 ml of water was added to arsenic Impingers 1 and 2 and 150 ml to Impingers 3 and 4. The maximum blank for the water corresponds to 3 μg for Impingers 1 and 2 and 2 μg for Impingers 3 and 4.

Laboratory reagent blank analyses were performed during the analysis of the field samples. The results of these analyses are presented in Table 3-3. The average value for five filter blanks was 29.7 µg out of a range of 26.7 to 32.8; a blank correction of 30 µg was used to correct all the reported data. All of the blank values for the rinse and impinger samples were near the analytical detection limit of 2 to 8 µg. Because of the variability and relatively small value of the blank, no average value was determined and no blank corrections applied.

Values below 50 μ g were considered insignificant and not reported because 8360 μ g (7570 on the filter) was the minimum amount of arsenic determined in any train and the blanks for the liquid sample fractions varied considerably.

Each sample was first analyzed by the flame technique. Samples whose concentrations were below 30 mg/liter were also analyzed using the graphite furnace. Actual sample concentrations were either greater than 100 or less than 10 ppm. The 30-mg/liter limit was based on previous experience with Method 108, which indicated good agreement above this level. As the analyses were completed and the data were reduced by the laboratory, the results were reviewed by the Quality Assurance Officer (QAO). The QAO reviewed instrument calibration, the analysis of the standard reference solution (SRS), agreement between flame and furnace results, and general consistency of the data. He then prepared a list of samples for reanalysis.

TABLE 3-3. ARSENIC LABORATORY REAGENT BLANK DATA

Date (1984)	Filter total, µg	Rinse, ^a mg/liter	Impingers, ^b mg/liter	Connector, ^C mg/liter
6/8	2.4	0.0079	0.0079	0.0079

 $^{^{}a}$ Between 235 and 328 ml of sample were received as the rinse fraction. The maximum laboratory reagent blank corresponds to 3 μg for this fraction.

 $[^]b$ Between 532 and 817 ml of sample were received as the Impingers 1 and 2 fractions and between 280 and 340 ml as the Impingers 3 and 4 fraction. These correspond to maximum laboratory reagent blanks of 6 μg and 3 μg , respectively.

 $^{^{\}text{C}}$ Between 119 and 180 ml of sample were received as the connector fraction. The maximum laboratory reagent blank corresponds to 1 μg for this fraction.

The analysis was performed in five batches by flame atomic absorption. Eighteen sets of standards (0, 10, 30, 50, 80, 100 ppm) were analyzed with the samples. The linear regression data for all the standards analyzed with a given batch of samples are presented in Table 3-4. The average correlation coefficient is 0.9989, out of a range of 0.9994 to 0.9985. The average detection limit is 2.3 ppm. A value of twice the range of the 0-ppm standard above the Y-intercept was used to calculate the detection limit. A standard reference solution independently prepared from AS₂O₃ with a nominal value of 150 ppm was analyzed (1-2 dilution) with each set of standards. (Standards were prepared from a commercially available 1000-ppm standard solution.) average value obtained in the 18 analyses of this standard reference solution (SRS) was 157.4 ppm, with a standard deviation (SD) of 3.81 ppm [2.4 percent relative standard deviation (RSD)]. Only 1 of the 18 determinations made fell outside the range of the mean ±2 SD (one was 166 ppm).

These data indicate that the precision and accuracy of the flame atomic absorption analyses are well within acceptable limits. The percent difference of the average measured value of the SRS and its predicted value is 4.9 percent; the RSD of the measured value is 2.4 percent.

The results of the audit samples supplied by EPA and determined by flame atomic absorption (listed in Table 3-5) are consistent with the data just presented. The relatively large difference at 10 ppm is predictable in that it is only 5 times the average detection limit.

TABLE 3-4. LINEAR REGRESSION DATA (FLAME)

Date (1984)	No. of standard curves	Y-intercept	Slope	Correlation coefficient	Detection limit, ppm
6/4	4	0.0031	0.00489	0.9990	2.1
6/5	2	0.0048	0.00500	0.9994	1.2
6/5	2	0.0056	0.00496	0.9985	1.6
6/11	5	-0.0011	0.00490	0.9990	1.6
6/13	5	0.0041	0.00450	0.9987	4.9

TABLE 3-5. ARSENIC AUDIT RESULTS

EPA No.	Lab No.	Volume, ml	Arsenic concentration EPA values, mg As/liter	Measured, mg/liter	Total As, mg
B-3-I	DC329	500	10	10.2	5.12
B-4-I	DC330	500	10	11.7	5.83
G-1-I	DC331	500	100	111	55.6
G-3-I	DC332	500	100	107	53.3
H-1-I	DC333	500	40	43.9	22.0
H-2-I	DC334	500	40	43.9	22.0
B-3-I ^a	DC329	500	10	11.1	5.54
B-4-I ^a	DC330	500	10	10.7	5.34
WP-475 ^a Conc 6	DM562	1000	0.207-0.393	0.356	0.356

^aGraphite furnace analysis.

Table 3-6 presents the results of 10 samples checked by the method of standard addition. The slopes of all the standard addition analyses are between 0.9 and 1.1 except for those of DM697, DM688, and DM660, which is probably due to an error in the spiking or the fact that no given point was in the regression analysis because a less-than value is unusable. An analysis of the results of the unspiked samples and the X-intercepts (standard addition values) revealed that only Sample DM697 showed a significant difference. The results for DM697 were expected, based on the slope; the results of standard addition show no consistent bias attributable to the sample matrices.

The samples were analyzed by atomic absorption in which graphite furnace techniques were used. All samples below 30 ppm were analyzed by furnace techniques. Sample concentrations were either greater than 100 ppm or less than 10 ppm. Values obtained from flame and furnace techniques cannot be accurately compared below 10 ppm because this value is too close to the flame detection limit. Twelve sets of standards (0, 0.01, 0.05, 0.10, and 0.15 mg/liter) were analyzed with the samples. All the data sets were reduced by linear regression analysis (see Table 3-7). The average correlation coefficient for the linear regression analysis was 0.9970, out of a range of 0.9980 to 0.9954. The average detection limit for the graphite furnace was 0.0033 ppm. A value of twice the range of the 0-ppm standard above the Y-intercept was used to calculate the detection limit.

TABLE 3-6. ARSENIC STANDARD ADDITION RESULTS

Lab	Number	Spike, ppm	Previously determined flame, ppm	Measured,	Linear regression analysis
DM643	Filter	0 20 30 40	26.16	22.45 45.58 52.03 62.70	Slope = 0.992 Y intercept = 23.36 Corr. = 0.9955 X intercept = -23.54
DM670	Filter	0 20 30 40	30.07	29.12 47.80 56.92 69.60	Slope = 0.995 Y intercept = 28.48 Corr. = 0.9974 X intercept = -28.64
DM697	Filter	0 20 30 40	31.45	35.35 48.47 57.37 66.26	Slope = 0.769 Y intercept = 34.55 Corr. = 0.9964 X intercept = -44.90
DM736	Filter	0 20 30 40	28.24	26.90 47.36 60.48 66.93	Slope = 1.030 Y intercept = 27.24 Corr. = 0.9957 X intercept = -26.45
DM637	Bomb	0 20 30 40	<2.6	<4.9 10.88 18.67 29.79	Slope = 0.946 Y intercept = 0.87 Corr. = 0.9949 X intercept = -0.92
DM688	Bomb	0 20 30 40	<3.5	<4.9 11.55 20.22 28.45	Slope = 0.845 Y intercept = 3.2 Corr. = 0.9999 X intercept = -3.76
DM650	Rinse	0 20 30 40	<2.9	<4.9 8.44 18.89 27.79	Slope = 0.968 Y intercept = 0.98 Corr. = 0.9989 X intercept = -1.01

(continued)

TABLE 3-6 (continued)

Lab Number	Spike,	Previously determined flame, ppm	Measured, ppm	Linear regression analysis
DM740 Rinse	0 20 30 40	5.5	<4.9 10.66 19.11 30.45	Slope = 0.990 Y intercept = 0.28 Corr. = 0.9965 X intercept = -0.28
DM660 Impinger	0 20 30 40	<2.1	<4.9 11.55 17.11 26.45	Slope = 0.745 Y intercept = 3.47 Corr. = 0.9894 X intercept = -4.66
DM717 Impinger	0 20 30 40	<1.3	<4.90 7.55 16.00 25.56	Slope = 0.900 Y intercept = 1.64 Corr. = 0.9994 X intercept = -1.82

TABLE 3-7. LINEAR REGRESSION DATA (FURNACE)

Date (1984)	No. of standard curves	Y-intercept	Slope	Correlation coefficient	Detection limit, ppm
6/8	2	-0.0011	4.081	0.9980	0.0039
6/11	4	0.0019	4.316	0.9975	0.0028
6/15	4	-0.0014	3.853	0.9954	0.0031

A standard reference solution independently prepared from As₂O₃ with a nominal value of 0.0750 ppm was analyzed with each set of standards. (Standards were prepared from a commercially available 1000-ppm standard solution.) The average value obtained for the 21 analyses of this SRS was 0.0751 ppm with a standard deviation of 0.0027 (3.6 percent relative standard deviation). Historically, the mean value for this SRS is 0.0762, with a standard deviation of 0.0027. The values obtained for the SRS solution during this project are in good agreement with our historical data. These data indicate that the precision and accuracy of the furnace atomic absorption analyses are well within acceptable limits. The difference in the average measured value of the SRS and its predicted value is 0.2 percent; the RSD of the measured value is 3.6 percent.

The results of audit samples analyzed using the graphite furnace were listed in Table 3-5. These values are consistent with the previous value and the accepted values. The results of duplicate analysis are presented in Table 3-8. The absolute value of the percent difference was calculated according to the following equation.

% Difference =
$$\frac{x_1 - x_2}{\bar{x}}$$

where \mathbf{X}_1 and \mathbf{X}_2 are the individual values \mathbf{X} is the average of \mathbf{X}_1 and \mathbf{X}_2

TABLE 3-8. DUPLICATE ANALYSIS DATA

Sample fraction	Arsenic, g	% Difference
Filter	8,310, 8,470 8,770, 9,130 9,690, 9,590 10,900, 11,100 10,900, 11,300 10,400, 10,300 10,800, 10,700 10,900, 10,400	1.4 3.0 1.0 1.8 3.6 1.0 0.9 4.7
Bomb	99, 93	6.2
Rinse	351, 294 498, 467 290, 269 610, 648 768, 657	17.7 6.4 7.5 6.0 15.6
Impinger	50, 48 4, 5	4.1 22.2
Filter ^a	36, 35	2.8
Bomb ^a	39, 32 61, 56 40, 28 66, 51	19.7 8.5 35.3 25.6
Rinse ^b	678, 693 623, 629	2.2
Impinger ^b	35, 30 32, 29 4, 49 227, 191 33, 30 5, 45 43, 39 153, 55	15.4 9.8 170 17.2 9.5 160 9.8 94

^aSame aliquot analyzed on different days.

 $^{^{\}mathrm{b}}\mathsf{Sample}$ aliquot prepared and analyzed on different days.

Filter samples were analyzed using flame atomic absorption, and all other sample fractions were analyzed using the graphite furnace technique.

The first 16 values reported are based on duplicate analysis of the same sample aliquot on the same day using the same calibration curves. The agreement on the front filters is very good. These filters contained better than 90 percent of the total arsenic collected. The average percent difference for the primary filter is 2.4 percent. The agreement for the other sample fractions is acceptable and will not have a significant influence on the overall method precision because they represent less than 10 percent of the total arsenic collected.

The next five values reported are based on repeat analysis of the same sample aliquot on different days using different calibration curves. The agreement is good considering the relatively small amount of arsenic contained in these fractions.

The last 10 values reported are based on repeat analysis of different sample aliquots prepared and analyzed on different days using different calibration curves. The agreement for the two rinse samples (the only two containing a significant amount of arsenic) is very good.

SECTION 4

SAMPLING LOCATION AND TEST METHODS

This section describes the sampling sites and test methods used to characterize arsenic emissions from each source evaluated.

A four-train (quad) sampling system was used to collect samples at the exit stack of the glass melting furnace. This system allows four trains to sample simultaneously at essentially a single point in the stack (see Figures 4-1 and 4-2).

Because this sampling approach allows four trains to sample simultaneously at essentially a single point, it reduces the effect of variations in the velocity and particulate profiles on the sampling results. It also permits a statistically significant number of samples to be taken in a short amount of time. Further, since all four trains are identical for every run, the within-train precision can be determined at the same time as the relationship of the different trains is being compared. This methodology for determining method precision was developed and validated in a previous EPA study.* A total of four quad-train runs representing 16 individual samples were collected. During

Mitchell, W. J., and M. R. Midgett. A Means to Evaluate the Performance of Stationary Source Test Methods. ES and T, 10:85-88, 1976.

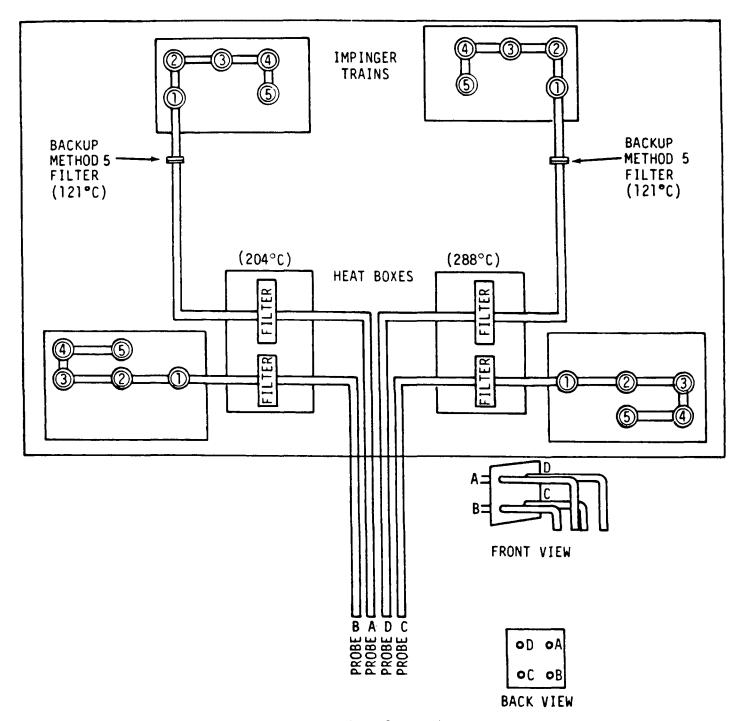


Figure 4-1. Quad train system for elevated temperature tests.

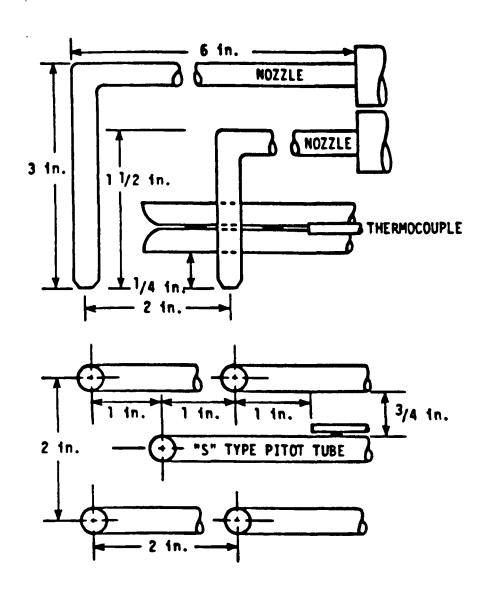


Figure 4-2. Four-train sampling system showing nozzle, pitot tube, and thermocouple position.

these runs, a single Method 108 train was run with the nozzle positioned as close to the quad nozzle arrangement as possible without causing interference.

All samples were collected at the furnace exit stack as depicted in Figures 4-3 and 4-4. Ambient ejector air is controlled automatically to maintain furnace pressure and is introduced angularly as depicted in Figure 4-3 at a volume ratio of about 1:1 to the furnace gases. According to plant personnel, the furnace gas temperature is about 760°C (1400°F), and exit gas temperatures ranged between 260° and 316°C (500° and 600°F), which indicated that the gases were relatively well mixed at the sample cross section. Single-point, isokinetic sampling techniques were used in each quad run and reference train tests. Prior to sampling, a complete velocity and temperature profile was established using procedures described in EPA Methods 1 and 2.* The velocity and temperature data were used to select sample nozzle sizes so as to maintain isokinetic sample rates and ensure adequate sample volume [0.85 dscm (30 dscf)] in each train. quad nozzle assembly was positioned approximately 52 cm (20.5 in.) from the inside wall of the stack in each run. Sampling rates generally ranged between 0.014 dsm3/m (0.50 dscf/m) and 0.017 dsm³ (0.60 dscf/m), and sampling times were typically 60 and 70 minutes.

In the Method 108 traverse tests, 24 sampling points were used to traverse the cross-sectional area of the stack. Each

^{*40} CFR 60, Appendix A, Reference Methods 1 and 2, July 1983.

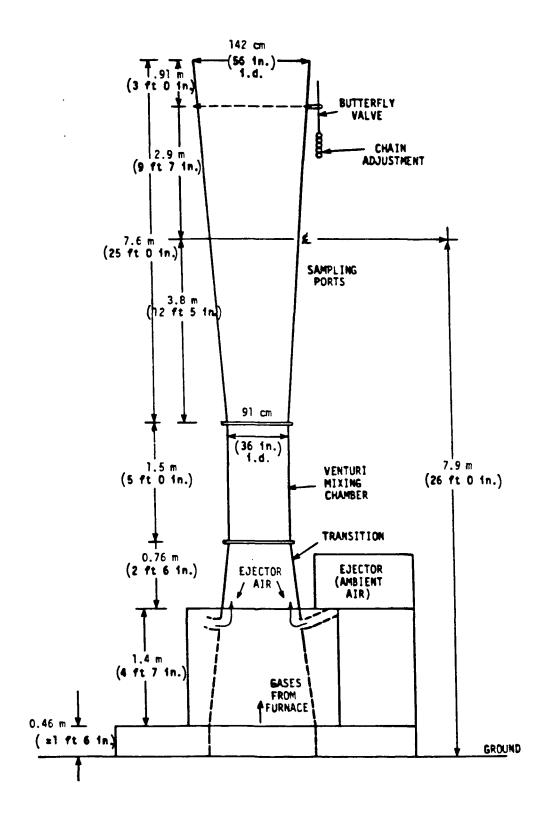
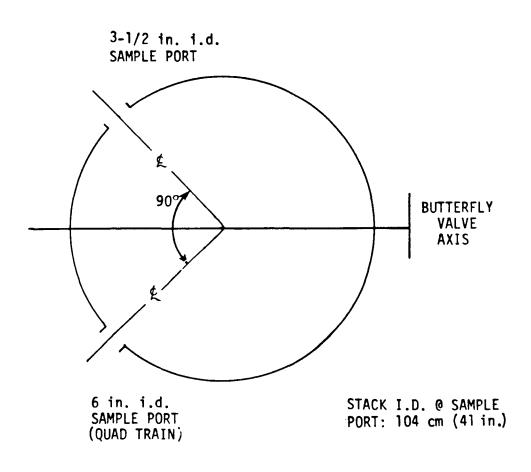


Figure 4-3. Furnace exit stack elevation (no scale).



CROSS SECTION

Figure 4-4. Furnace exit stack sampling port location (no scale).

point was sampled for 2.5 minutes, yielding a total test time of 60 minutes.

4.1 SAMPLING AND ANALYTICAL PROCEDURES

The sampling and analytical procedures used in this test program followed those described in EPA Reference Methods 1 through 4* and proposed Method 108 as detailed in the site test plan prepared by PEI and reviewed by EMB. The procedures, which are described briefly here, are detailed in Appendices D and F.

4.1.1 Velocity and Gas Temperature

A Type-S pitot tube and an inclined draft gauge manometer were used to measure gas velocity pressures at the test sites. Temperature was measured with a thermocouple and digital readout. During each sample run, velocity and temperature measurements were taken at a single sampling point in the duct. Prior to each test series, separate velocity measurements were taken by traversing the entire sample cross-sectional area to determine an average value. Measurements were taken in accordance with procedures outlined in Reference Method 2 of the Federal Register.*

4.1.2 Molecular Weight

Flue gas composition was determined in accordance with the basic procedures described in Reference Method 3.* Grab samples were collected before any sampling began to establish baseline contents of oxygen, carbon dioxide, and carbon monoxide. Bag

 $^{^{\}star}40$ CFR 60, Appendix A, Reference Methods 1 through 4, July 1983.

samples were collected at least twice daily during sampling at each source and analyzed with an Orsat gas analyzer. An integrated bag sample was also collected during each Method 108 traverse test and analyzed using an Orsat gas analyzer.

Method 108* was used to measure arsenic concentration except that the impingers containing hydrogen peroxide (H2O2) for SO2 determination were eliminated due to low (less than 30 ppm) concentrations of SO2. All tests were conducted isokinetically by regulating the sample flow rate relative to the gas velocity in the stack as measured by the pitot tube and thermocouple attached to the quad probe arrangement (see Figure 4-2). Each individual sampling train consisted of a heated glass-lined probe, a heated 7.6-cm (3-in.) diameter glass fiber filter (Whatman Reeve Angel 934AH), and a series of four Greenburg-Smith impingers followed by a vacuum line, vacuum gauge, leak-free vacuum pump, dry gas meter, thermometers, and a calibrated ori-In each train, probe and filter temperatures were monitored using digital indicators and thermocouple leads located in each probe and immediately behind the Method 108 filter frit. Quad Runs 10, 12, and 13, a 53-cm (21-in.) glass connector was used to attach the front filter to a backup filter maintained at approximately 121°C. The impingers followed the backup filter in these runs.

The amount of water collected in the impinger section of the sampling train was measured gravimetrically at the end of each

^{*}Method 108 is proposed. 40 CFR 61, Appendix B, Method 108, July 1983.

sample run to determine the moisture content of the flue gas. The contents of the first two impingers, each of which had been charged initially with 150 ml of distilled water, were transferred to a polyethylene container. These impingers and all connecting glassware (including the back half of the filter holder) as well as a third (empty) impinger were rinsed with 0.1 N NaOH; the rinse was then added to the container. The contents of the first two impingers and 0.1 N NaOH rinse were analyzed for arsenic by atomic absorption. In the elevated temperature runs, the third and fourth impingers were recovered and analyzed similar to Impingers 1 and 2.

SECTION 5

PROCESS OPERATION

Tests were performed on the uncontrolled emissions from a regenerative natural-gas-fired glass melting furnace. The furnace evaluated has a pull-rate capacity of 90 to 100 tons/day and produces primarily crystal glass utilizing arsenic as a conditioning and refining agent. Furnace pressure is maintained by use of an induced-draft ejector system as described in Section 4. Data collected during this study indicate that the furnace gases and the ejector air were adequately mixed at the sampling location.

Personnel from Radian Corporation (an EPA contractor) monitored the furnace operation during each test. Appendix F of this report contains a detailed process description and a summary of furnace operating data.